


## Responses to California Department of Public Health (CDPH) Technical Questions on Sr Exceedance Reports and RSY Data Reports

Comments by: CDPH, comments received August 13, 2021

Comments	Response
<p><b>1. Hunters Point Naval Shipyard, PARCEL G TU79 and TU124 Report, Page 2, 4th Paragraph, Sentence 6:</b> “The soil at HPNS is known to be highly heterogenous.” Please provide a reference or description that supports the statement above.</p>	<p>From the Parcel G Record of Decision (ROD; Navy, 2009): “Parcel G consists of flat lowlands that were constructed by placing borrowed fill material from various sources, including crushed serpentinite bedrock from the adjacent highland and dredged sediments with surface elevations between 0 to 10 feet above mean sea level.”</p>
<p><b>2. Hunters Point Naval Shipyard, PARCEL G TU79 and TU124 Sr Exceedance Reports Page 2, 4th Paragraph, Sentence 6:</b> “The soil at HPNS is known to be highly heterogenous. A sample can be homogenized per the laboratory standard operating procedures and still be heterogenous, as demonstrated from the results included in Table 2.” Please explain detail steps of your laboratory standard operating procedures for homogenization of soil samples.</p>	<p>The samples are dried in a drying oven for a minimum of 8 hours and then rolled on the ball mill for a minimum of 8 hours. Pictures below show a before (site sample) and after lab drying/tumbling (these examples are provide for a visual and not specific sample TU79 samples).</p> <div data-bbox="1266 665 1806 1102" data-label="Image"> </div> <p style="text-align: center;">Site Sample</p>

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	 <p>After Drying and Tumbling in ball mill.</p> <p>The aliquots taken for analysis from this process are heterogenous to the extent possible.</p>
<p><b>3. Hunters Point Naval Shipyard, Parcel G, TU79 Analytical Report, Section: Case Narrative, Page 4 of 35,</b> “When taking small mass aliquots from dried/disaggregated sample, the laboratory avoids large rocks/pebbles (as well as sticks, etc) which may constitute a larger than representative portion of the aliquot. Smaller rocks may be included.” Did these debris, large rocks, pebbles, or sticks, get scanned for radiation or be processed as part of the sample?</p>	<p>This is generic soil sample preparation language and not specific to the samples in this group. The note is indicating that after drying/tumbling in the ball mill most of the soil sample is disaggregated into a fine mixed soil sample, however the ball mill does not crush small rocks or twigs into powder. Therefore, when taking small aliquots out for analysis, the chemist take the fine/mixed soil for analysis and avoid small rock or twigs that may be present. Before a sample leaves the site, a radiation/dose rate survey is conducted.</p>
<p><b>4. Hunters Point Naval Shipyard, Parcel G, TU79 Analytical Report, Section: Case Narrative, Page 4 of 3:</b> “When taking small mass aliquots from dried/disaggregated sample, the laboratory avoids large rocks/pebbles (as well as sticks, etc) which may constitute a larger than representative portion of the aliquot.” Please provide the typical weight of the material used before the chemical separation and the weight of the final material used for GFPC counting. If the data is available, please provide the same information in the Hunters Point Background Study (June 2020).</p>	<p>Eurofins-TA uses 1 gram for Sr-90 analysis and when counting for Sr-90, the mass of the yttrium oxalate on the planchet is approximately 30 mg.</p> <p>This specific information from the background study is unknown.</p>
<p><b>5. Hunters Point Naval Shipyard, Parcel G, RSY Data Report:</b> According to those data shared by Navy, Sr-90 results from Parcel G soil have</p>	<p>APTIM laboratory Eurofins-TA follows a different sample preparation procedure than the background study laboratory GEL, which accounts for</p>

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<p>uncertainty values that are consistently higher than those obtained from the RBAs in HPNS Background study. Please explain the reason for the higher uncertainty from Parcel G and all possible factors, including but not limited to count time, ingrowth period, aliquot weight, etc. At the same time, please provide the equation for calculating uncertainty, including the description of all the parameters used for uncertainty calculation</p>	<p>the difference in total uncertainty. Eurofins-TA does have a method that more closely mimics the procedure used by GEL during the background study. The current Eurofins-TA method uses a 1 gram aliquot with a 7-day ingrowth period.</p> <p>The proposed Eurofins-TA method (that closely mimics the GEL procedure) uses a larger aliquot (2.5 grams) with HNO<sub>3</sub>/HCl digestion and Eichrom resin (Sr Resin) separation, a 14-day ingrowth period, and gas flow proportional counter (GFPC) detection. Based on Eurofins-TA's experience with this method, the laboratory expects to see lower DLC and total uncertainty.</p> <p>Eurofins-TA is certified with DoD and DOE for this preparation method for Sr-90 detection. Field Change Request (FCR)-006 is under consideration with the Navy which would allow APTIM to begin using this method.</p> <p>The laboratory uncertainty calculations are provided in Eurofin-TA's Quality Assurance Manual (excerpt is provided the attached PDF).</p>
<p><b>6. Hunters Point Naval Shipyard, Parcel G, RSY Data Report:</b> The method blank for the 4 additional aliquots at TU79 (prep batch 495823) yielded a Sr-90 results higher than LOQ. Please provide the explanation on how the method blank samples are created and the possible reasons for elevated of Sr-90 concentration in a method blank sample.</p>	<p>The method blank is a clean, matrix equivalent material (sand) created by the laboratory that is processed simultaneously with, and under the same conditions as samples through all steps of the analytical procedures. The exact cause of the blank detection in the prep batch is unknown. As stated in MARLAP, Ideally, no target analytes should be present in the blank at detectable concentrations. However, MARLAP further states that the results from blanks can either be positive or negative.</p>
<p><b>7. Hunters Point Naval Shipyard, Parcel G, RSY Data Report:</b> The method blank for the 4 additional aliquots at TU79 (prep batch 495823) yielded a Sr-90 results higher than LOQ. MARLAP states that; "Blank samples are used to determine whether any radionuclide contamination is introduced by the measurement process. Ideally, no target analytes should be present in the blank at detectable concentrations." Please describe how the method blank is used to determine if there is any QC issue of the measurement. At the same time, please explain why the Sr-90 result of the method blank in prep batch 495823 being higher than LOQ did not cause any QC concern in the measurements of the 4 additional aliquots samples.</p>	<p>As stated in MARLAP, ideally, no target analytes should be present in the blank at detectable concentrations. MARLAP further states that the results from blanks can either be positive or negative. Method blanks (MB) are used to evaluate bias in the measurements. Although the MB detection does indicate some potential contamination bias in the analytical batch, the associated sample results were all not detected (U). Therefore, a "potential contamination indicated by the MB" did not affect project sample results, and therefore did not affect the usability of the data.</p>

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The evaluation of method blank data relative to sample results is described in the Project Approved SAP WS#36.

Method Blank	Blank < MDC	unusable (R)	Associated analytes in all samples in preparation batch or analytical batch
		None required	
	Blank > MDC	Sample < MDC: None required	
		Sample > MDC by < 10x blank, qualify as estimated (J)	
		Sample > 10x blank, None required	
		Sample > MDC: qualify as	

IF MB results are above LOQ and associated sample results were above LOQ, this would trigger reanalysis of the analytical batch, since the method blank detection would interfere with evaluation of sample results and decisions could not be made using data from the batch.

**8. Hunters Point Naval Shipyard, Parcel G, RSY Data Report (HPPG-ESUTU79B-B-001, HPPG-ESU-TU153C-001):** "The method blank (MB) Z-score is within limits and is located in the level IV raw data. (MB 160-493207/23-A)." Please explain acceptable limits of MB Z-score, numerical value of the level IV raw data and Z-score of the method blank sample. Please also explain the reason that Z-score is only provided for these two MBs.

The MB Z-score is a requirement in MARLAP. The z-score, or z factor, is a measurement of the blank value divided by the combined standard uncertainty (CSU). The Z-score control limit is <3. The method blank Z-score is provided for every analytical batch in every Level IV Data package. Level IV data packages are large files (typically 17-20 MB each) and are included in the Final Remedial Action Completion Report (RACR).

**9. Hunters Point Naval Shipyard, Parcel G, RSY Data Report:** Some of the calculated RER values in the analytical data reports (i.e. HPPG-ESU-TU077A-015) fail the criteria ( $\geq 1$ ). Please explain how analytical data validation and duplicate sample results were assessed/flagged if RER values fail the criteria.

(SDG J40423) RER values >1 for sample HPPG-ESU-TU077A-015 are for Thorium-234 and Uranium-238 by Gamma Spec. Gamma Spec is used to report and evaluate Ra-226 and Cs-137 only; the RER for these analytes passed. All other Gamma Spec nuclides reported are standard TA report format and this data are not used, validated or reported for Parcel G. If Thorium/Uranium is an ROC for the trench and is needed for evaluation, these ROCs are analyzed using Alpha Spec, not Gamma Spec.

**10. Hunters Point Naval Shipyard, Parcel G, RSY Data Report:** A duplicate sample (DU) analysis and RER calculations for Sr-90 are included in some of the analytical results but not provided in most of the laboratory reports. According to TestAmerica SOP No. ST-RC-0050, Rev 18 provided in the Appendix B, Attachment 2, 1\_TA\_SOP, "A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents." and "For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), and Sample Duplicate. In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed." To CDPH's

An analytical batch is not the same as a sample delivery group (SDG). The analytical batch for Sr analysis may include samples from multiple SDGs which are grouped together into an analytical batch (up to 20 field samples). One sample is selected for DU analysis and is reported with its applicable SDG, therefore a DU for Sr-90 may not appear in each lab report, however, the required batch frequency has been met. This can be tracked by "preparation batch number" in the individual SDGs.

For data packages going forward, the laboratory will include the batch QC (DU report) in every SDG for all samples in a preparation batch.

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understanding, SOP No. ST-RC-0050 states that a Sample Duplicate (DU) should be prepared for a batch (maximum of 20 environmental samples) that would get analyzed for Strontium concentration. In contrast, TestAmerica only created a DU for every batch (maximum of 20 environmental samples) that got analyzed for various ROCs. Since Strontium analysis is performed on only 10% of the samples that got analyzed for various ROCs, there was not a DU created for every batch, which can consist 1 to 20 samples, intended for Strontium analysis. In addition, no LCSD was created for those events that there were insufficient sample to analyze a sample duplicate. Please explain how the quality control can be measured or assessed on a batch of samples intended for the Strontium analysis that was not processed with a DU.

**11. Hunters Point Naval Shipyard, Parcel G, RSY Data Report:** "The laboratory control sample (LCS) associated with the following samples falls below the lower limit for spike criteria (recovery is 74%; criteria is 75-125%): HPPG-317364365-SU28C-001 (160-40591-1), (160-39992-A-30-C) and (160-39992-A-30-D DU)." Since a matrix spike test increases confidence in the accuracy and validity of the sample measurement process. Please explain why the recovery yield being lower than the criteria range did not raise any QC issue.

A 1% low recovery in an LCS would not constitute an entire analytical batch failure. And would not invalidate the associated sample data use. This would be considered a "minor exceedance" and was still within the laboratory statistically derived control limits for this method. This minor exceedance would be handled during data validation process and all QC samples in the batch would be evaluated against project sample results.

SAP WS#36

LCS	%R >UCL	Sample > MDC, qualify as estimated (J)	Associated analytes in all samples in preparation batch or analytical batch
	%R <LCL but ≥ 30%	Sample < MDC, qualify as estimated (J) Sample < MDC, qualify as estimated (JJ)	

**12. Hunters Point Naval Shipyard, Parcel G, RSY Data Report:** In the HPNS Parcel G Rework WP, a footnote (10) in Worksheet #14 specifies that; "reported radiological results will, at a minimum, include count times, results, counting uncertainty, and total propagated uncertainty." However, the count time is not included in all the data and soil analytical reports. Please include the count time in all the previous and future analytical data reports.

Count times are provided by the lab in all Level IV data reports which are included in the Final RACR.

**13. PARCEL G TU 79 Report page 2, 6th Paragraph 2th Sentence:**  
"Following a 7-day yttrium-90 (90Y) (90Sr daughter product) ingrowth

The laboratory was following their SOP and historic practices for Hunters Point by using a 7-day ingrowth period. The ingrowth equilibriums are

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period to approach secular equilibrium with <sup>90</sup>Sr, the <sup>90</sup>Y is precipitated from the aliquot and plated on the planchet.” Please explain why a 1-week ingrowth time period was deemed sufficient for Strontium measurement.

well documented and after a 7-day ingrowth period yttrium-90 is at 83.7% equilibrium. The final sample results are corrected for using the exact ingrowth period by the following equation from the Eurofins-TA Quality Assurance Manual (page 157 of 169):

Sr-90 Ingrowth/Decay factors:

$$I = (1 - e^{-\lambda t_1})(e^{-\lambda t_2})$$

Where:

I = Ingrowth Factor

$\lambda$  = ln(2)/Y-90 Half-life (in days)

Y-90 Half-life = 2.67 days

t1 = Time between Strontium Precipitation Time and Yttrium Precipitation Time

t2 = Time between Yttrium Precipitation Time and CountMidPoint

**14. ANALYTICAL REPORT, HPPG-ESU-TU079A-001 with 4 Additional Aliquots\_TU79, Page 4:** “A sample duplicate (DU) was not reported for this batch due to the client requesting 4 replicates of this sample to be reported HPPG-ESU-TU079A-001 (160-40475-1).” Please explain, if a duplicate sample was created and measured together with 4 replicates. If so, please provide the result of this DU sample. Similarly, please explain if a duplicate sample was created and measured together with the HPPG-ESU-TU079A-001 sub-sample yielded 0.334 pCi/g, and analyzed on 12/10/2020. If so, please provide the result of the DU.

A laboratory duplicate (DU) is prepared by taking two separate aliquots from the dry/homogenized sample processing them simultaneously with, and under the same conditions through all steps of the analytical procedures, and analyzing them side by side. For the quadruplicate re-analysis process, the laboratory took four separate aliquots from the dry/homogenized sample processed them simultaneously with, and under the same conditions through all steps of the analytical procedure. Therefore, no additional laboratory duplicate analysis was performed on this analytical batch, the sample was analyzed in quadruplicate, four additional aliquots prepared and analyzed together.

**15. ANALYTICAL REPORT, HPPG-SFU-TU107A-B-001:** “The replicate precision (RER) for Th-234/U-238 does not meet QC criteria. This appears to be random in nature, and limited deviations such as this are statistically expected when larger analyte lists are reported. Such excursions are often caused by fluctuations in Compton background, force-fitting of peaks that are not found by the software peak-search algorithm, and inclusion of inferior peak results by the software in weighted averages. The laboratory SOP allows for such statistical exceedances.(160-40595-A-5-C DU). Please provide a reference/description that supports the statement above.

HPPG-SFU-TU107A-B-001 is reported in SDG 160-40597-1, There is no RER reported in this SDG, no mention of RER in the narrative.

The note at the end of this comment is in reference to SDG 160-40595. This note refers to Gamma Spec (Thorium and Uranium) see response #9.

Where:

CPM = counts per minute (S=Sample, B=Background, XT=crosstalk, α=alpha)  
T=count duration in minutes (S=Sample, B=Background)  
E =Efficiency  
V =aliquot volume  
UF =uncertainty factor (e.g. 0.05)  
Act =activity

### RadCapture Equations

RadCapture, an in-house developed software, is utilized for calculation of results for all methods except gamma spectrometry (which uses GammaVision). All equations use the general form:

$$Activity = \frac{\left( \frac{C_s}{T_s} - \frac{C_{xt}}{T_s} - \frac{C_b}{T_b} \right)}{D * E * I * V * R * A} * DF * UCF$$

$$UncCnt (1\sigma) = \frac{\sqrt{\frac{C_s}{T_s^2} + \frac{C_{xt}}{T_s^2} + \frac{C_b}{T_b^2} + Chi^2}}{D * E * I * V * R * A} * DF * UCF$$

$$UncTot (1\sigma) = \sqrt{UncCnt^2 + (IPUFact * Activity)^2}$$

$$MDC = \left( \frac{3.29 \sqrt{\frac{C_b}{T_b * T_s} + \frac{C_{xt}}{T_s^2} + \frac{C_b}{T_b^2} + Chi^2}}{D * E * I * V * R * A} + \frac{3}{D * E * I * V * T_s * R * A} \right) * DF * UCF$$

$$DLC = \left( \frac{1.645 \sqrt{\frac{C_b}{T_b * T_s} + \frac{C_{xt}}{T_s^2} + \frac{C_b}{T_b^2} + Chi^2}}{D * E * I * V * R * A} \right) * DF * UCF$$

Where:

Cs = Sample Counts  
Cb = Background Counts  
Cxt = Crosstalk Counts (interference correction)  
Ts = Sample Count Duration  
Tb = Background Count Duration  
D = Decay  
E = Efficiency  
I = Ingrowth

Facility Distribution No. \_\_\_\_\_

Distributed To: \_\_\_\_\_

V = Aliquot Volume  
R = Recovery  
A = Abundance (Branching Ratio)  
DF = Dilution Factor  
UCF = Units Conversion Factor  
Chi = non-Poisson variance

For the count uncertainty, if both Cs and Cb = 0, then 1 is forced into Cs.  
For the DLC, if Cb = 0, then 1 is forced into Cb.

RadCapture Tals allows for interference corrections, which are applied through the "crosstalk" (Cxt) factor. This calculation is consistent with interference corrections discussed in MARLAP chapters 19 and 20, where the interference factor is shown as  $R_i$  (count rate of the interference) in equations 20.6 and 20.7. In the laboratory equations this factor is CPMxt (or Cxt/Ts). Interference corrections may be applied on a batch by batch basis, and are normally performed upon client request, when lower detection criteria are needed (small interferences become significant at lower levels), or when the interference is more pronounced.

Interferences may include, but are not limited to, impurities in reagents, tracers, or glassware such as naturally occurring isotopes of uranium, thorium, and radium. It can also account for interferences such as tailing of Th-229 into the Th-230 region of interest (ROI) or for impurities seen in tracers/carriers. Acceptable means of determining interference correction factors may include the use of manufacturer certificates or a blank population type study.

The non-Poisson variance (Chi) is available for client-specific needs. It is included for all methods to create consistency in the calculation equations. This factor is based upon non-Poisson variances as discussed in MARLAP chapters 19 and 20, and outlined in equations 20.6 and 20.7.

#### **Equations for Isotopes by Mass and Activity ICP-MS (Uranium by Mass)**

Facility Distribution No. \_\_\_\_\_

Distributed To: \_\_\_\_\_